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ВЛИЯНИЕ СОДЕРЖАНИЯ НИОБИЯ НА МИКРОСТРУКТУРУ И МЕХАНИЧЕСКИЕ СВОЙСТВА СПЛАВОВ Ti–Nb ДЛЯ ПРИМЕНЕНИЯ В МЕДИЦИНЕ

В работе были систематически исследованы двухкомпонентные сплавы системы Ti–Nb. Количество Nb, используемого в качестве легирующего элемента, варьировалось от 0 до 37 вес. %. Все сплавы были сформированы методом дугового переплава в инертной атмосфере аргона. Полученные результаты свидетельствуют о том, что при содержании легирующего элемента более 37 вес. % в сплавах формируется β-фаза. Максимальная твердость (393 ± 10 HV0.05) была достигнута в сплаве, содержащем 24 вес. % Nb после отжига.

Ключевые слова: биоматериалы, титановые сплавы, микроструктура.

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THE INFLUENCE OF NB CONTENT ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Ti–Nb ALLOYS FOR BIOMEDICINE

The effect of Nb content on microstructure and mechanical properties of binary Ti–Nb alloys has been studied. The alloying content of Nb varied from 10 wt % to 37 wt %. All alloys were produced by arc melting in inert argon atmosphere followed by annealing and quenching. The results indicate that a stable β-phase was formed above an alloying content of 37 wt %.

Keywords: TI–NB alloy, metallic biomaterial, microstructure.

Ti and its alloys are widely utilized as biomaterial because of their good biocompatibility, high mechanical properties and enhanced corrosion resistance. The main issue of Ti based biomaterials are low shear strength and a relatively high Young`s modulus [1, 2]. A significant surge in the use of Ti and its alloys started in the early 1950`s. Since that time Ti–Alloys are exploited as materials for biomedical applications. The most widely used Ti–Alloy in medicine is Ti–6Al–4V [3]. Besides Ti–6Al–4V stainless steel or Cr–Co alloys are in service [4]. All above mentioned materials have relatively high Young`s moduli in common. The Young`s modulus of a human bone is in the range 4 –40 GPa, whereas the modulus of Ti–6Al–4V is around 110 GPa [5]. Stainless steels and Cr–Co alloys possess the modulus above 200 GPa. The big difference in

Young`s modulus between a medical implant and a human bone causes an effect called stress shielding, which leads to subsequent loosening of the implants [6]. One possibility to avoid stress shielding effect is the use of metallic alloys with lower Young`s modulus. Another drawback of the aforementioned alloys is their low biocompatility. Alloying elements such as chromium, nickel or cobalt can be the reason for allergies which leads to rejection of the implant. Aluminium can be the reason for Alzheimer`s or Parkinson`s diseases whereas vanadium is known as rather toxic element for human body [7]. The purpose of this study is to investigate the effect of Nb content on the microstructure, phase composition and some mechanical properties of binary TI–NB alloys, which are currently considered to be promising candidates to replace currently used alloys due to their low Young`s modulus and perfect biocompatibility.

The ingots made of commercially pure (c.p.) Ti and binary TI–NB alloys with a Nb content between 0 wt. % and 37 wt. % (hereafter wt. % will refer to as %) were prepared in an BUHLER arc furnace. Prior to melting the furnace was evacuated and flushed with argon three times. An argon pressure of 800 mPa was maintained throughout melting. For melting a water-cooled copper hearth and a non-consumable tungsten electrode were used. Considering the big difference in melting temperature (Ti:1941 K; Nb 2750 K) and density (Ti: 4,5 g/cm³; Nb: 8,57 g/cm³) between the initial materials and the relatively broad two-phase field in the TI–NB phase diagram the samples were melted 16 times and flipped after every second melt. Prior to each melting procedure a c.p. Ti ingot was melted as an oxygen getter. This was done due to the relatively high reactivity of Ti and Nb with oxygen at elevated temperatures. The initial materials and as-melted button shaped samples were weighted to observe the possible weight loss during the melting. All ingots were homogenized in vacuum at 1000 °C for 24 h to eliminate the as-cast microscopic segregations and then cooled in the furnace to room temperature. The homogenized ingots were subjected to a solution treatment at 1000 °C, which is above the β transus temperature, for 0.5 h followed by a rapid quenching in oil. After the quenching the ingots were cut into several pieces, which were embedded into epoxy resin. The samples were mechanically ground using SiC paper up to grade 1000, followed by pre-polishing with Al_2O_3 powder. Final polishing was conducted using colloidal SiO_2. The samples were etched using a Kroll`s solution consisting of 2 vol. % HF, 6 vol. % HNO_3 and 92 vol. % distilled water. Microstructures of the samples were observed by optical microscopy (OM) using a Carl Zeiss AxioObserver Z1m microscope and scanning electron microscope (SEM) using a Carl Zeiss EVO 50 microscope. The elemental composition was checked by energy dispersive X-ray spectroscopy (EDX) Oxford Instruments X-Act coupled with SEM. The Vickers hardness test was used to evaluate the mechanical properties. At least 30 measurements with a load of 50 g and a dwell time of 10 s were done with a WOLPERT Group 402 MVD Vickers hardness tester to achieve a significant average. X-ray diffraction analysis was implemented using an ARL X`TRA diffractometer using Cu k$\alpha_{1,2}$ radiation in 2Θ range from 30° to 80°. The various phases were identified by matching each characteristic peak from the samples with the International Centre for Diffraction Data (ICDD) PDF4 database.

The weight losses were found to be between 0.01 and 0.83 % which indicates that the actual composition of each alloy is close to the nominal composition. EDX analysis of the binary TI–NB alloys were carried out to check the actual elemental composition of the alloys. The results are shown in Table 1. XRD analysis revealed that the annealed samples containing up to 25 % of Nb consist of hcp α`, whereas the samples with a Nb content above 25 % are composed of β phase. After the quenching the samples consist of martensitic α``and β phases. When an alloying content of 37 % is reached stable β is obtained. It is well accepted that only two stable solid phases exist in TI–NB alloys, namely hcp α phase and bcc β phase. Besides them four nonequilibrium phases: martensitic α`, α``, ω and metastable β can exist. The formation of the martensite in TI–NB strongly depends on the Nb content as well as on the cooling rate during quenching. The ω phase with hcp structure can be formed during quenching the sample from a temperature in the β field or during ageing [8]. The former phase refers as ω_{ath}, while the latter is mentioned as ω_{iso}.

Table 1

Elemental composition and mass loss of researched samples

Sample	Nb, %	Ti, %	Relative mass loss, %
c.p. Ti		100	
Ti–15Nb	14 ± 0.7	Balance	0.83
Ti–25Nb	24 ± 0.8	Balance	0.10
Ti–30Nb	29 ± 0.2	Balance	0.13
Ti–35Nb	34 ± 0.5	Balance	0.01
Ti–37Nb	37 ± 0.4	Balance	0.04

Fig. 1a shows that the cast c.p. Ti exhibit a typical lath type morphology. Fine acicular α` martensite is visible in the sample with an alloying content of 25 % Nb after the annealing. α` colonies are localized at the grain boundaries and growing from the grain boundaries into the β matrix. Fig. 1c shows the same sample after the quenching. The structure consists of two phases: martensite and β phase. The volume fraction of the martensite decreases with increasing the Nb content. Long relatively thick primary martensitic plates with and without distinct boundaries are visible. When the alloying content reaches 37 % a typical β structure is obtained.

Fig. 2 shows the result of the microhardness measurements. All samples exhibit a higher microhardness than c.p. Ti. The microhardness of the annealed samples increases up to a maximum of 393 ± 10 $HV_{0,05}$. This microhardness value corresponds to the sample with 29 % Nb. With further increasing the alloying content the microhardness decreases again. It is suggested that this peak in microhardness is due to the presence of ω phase. Mantani and Tajima [9] showed that 1h of aging treatment above a temperature of 573 K is enough to receive metastable ω phase in TI–NB alloys in an alloying range between 25 and 40 %. The maximum microhardness of quenched samples is 246 ± 6 $HV_{0,05}$, which corresponds to the Ti–15Nb sample.

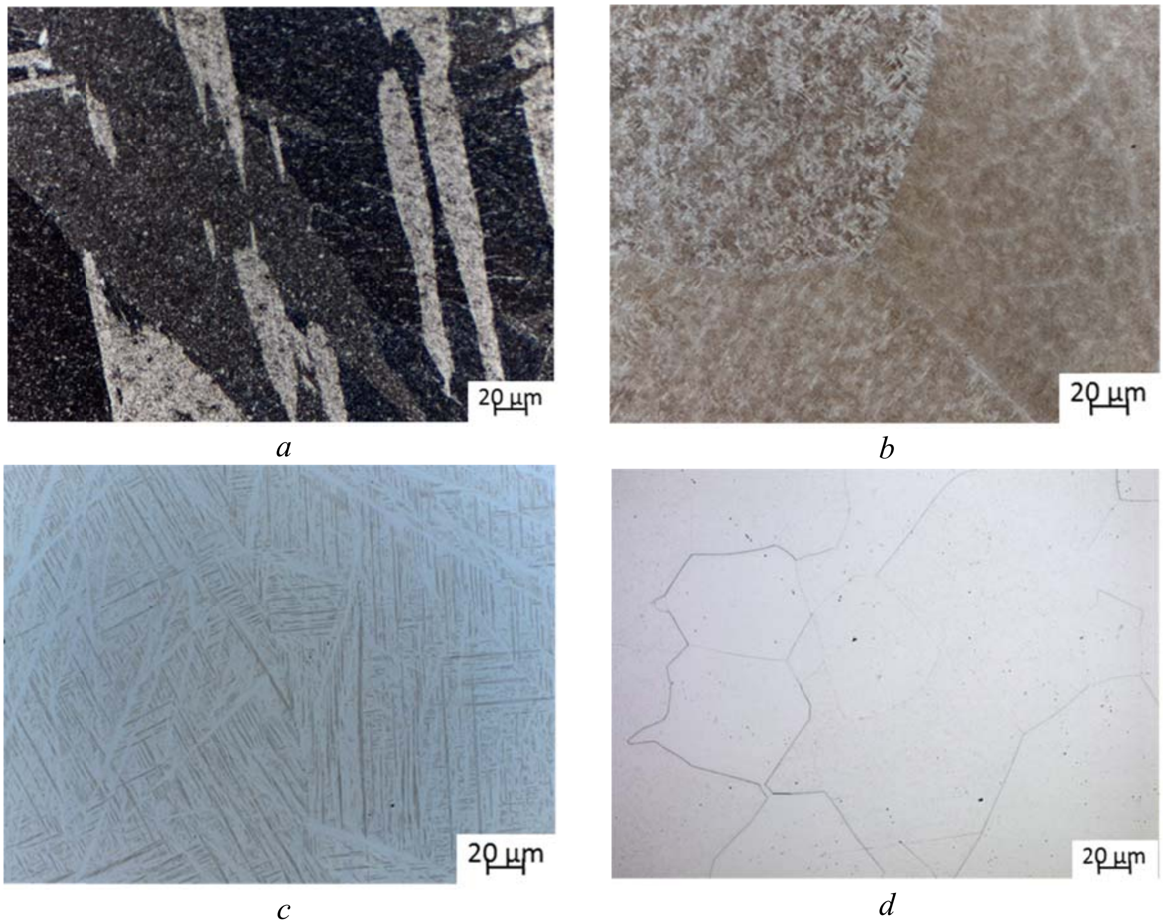

Fig. 1. Light microscopy *a* – c.p. Ti, *b* – Ti–25Nb after annealing, *c* – Ti–25Nb after quenching, *d* – Ti–37Nb after quenching

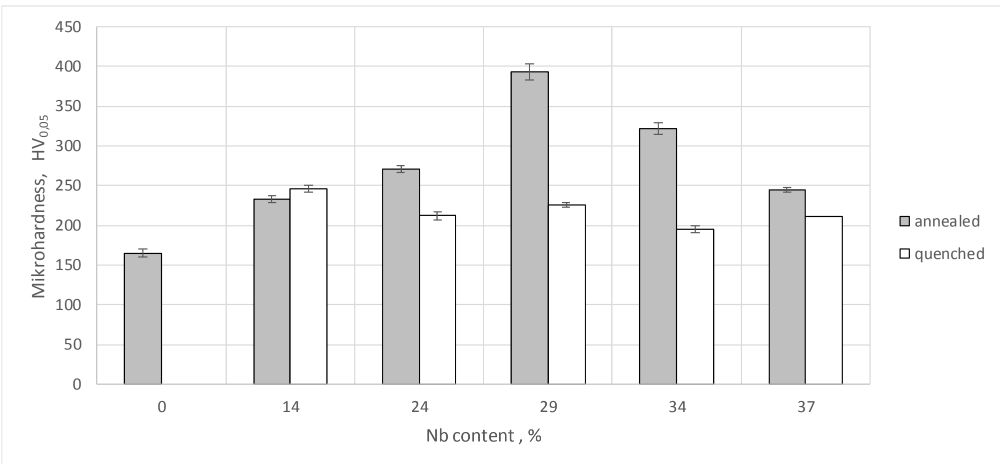

Fig. 2. Microhardness of c.p. Ti and TI–NB alloys

Experimental results indicated that both the microstructure and the mechanical properties strongly depend on the Nb content After annealing α` and β were predominating phases, after quenching the alloys consist of of α`, α`` and β phase. All samples showed a higher microhardness than c.p. Ti. The microhardness of alloys with metastable ω is higher than that of alloys with nonequilibrium α` and α`` phases. The lowest hardness possessed alloys were β phase was the dominating phase.

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